

MR 280349



October 26, 2004

8EHQ-1004-15976\$

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Office of Pollution, Prevention and Toxics
U.S. Environmental Protection Agency
1200 Pennsylvania Avenue, N. W.
Washington, DC 20460
Attention: Section 8(e) Coordinator

Re: **TSCA Section 8(e) Submissions**

Dear Sir/Madam:

3M Company ("3M") requests that EPA place the attached studies in the TSCA Section 8(e) docket. We have included a master index for these studies identifying the study title, test substance and CAS number. A Confidential Business Information (CBI) version of this index and the studies also is being submitted today pursuant to EPA procedures. 3M has not provided CBI substantiation with this submission, but would be willing to do so at the Agency's request.

3M has concluded that data in these studies may not be, strictly speaking, "corroborative" of previously reported or published information as defined in EPA's reporting guidance or otherwise potentially may warrant 8(e) submission based on EPA's reporting guidance.

3M appreciates EPA's attention to this matter. Please contact the undersigned if you have any questions or require further information regarding this submission.

Very truly yours,



Katherine E. Reed

Dr. Katherine E. Reed, Ph.D
Staff Vice President
Environmental Technology and Safety
Services
(651) 778-4331
kereed@mmm.com

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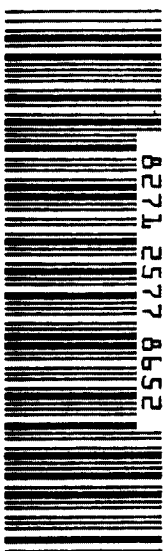
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4a Express Package Service

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FedEx Standard Overnight

FedEx First Overnight

Next business morning

Delivery commitment may be later in some areas.
Federal Express International
Delivery to select locations.

4b Express Freight Service

☐ FedEx 2Day

FedEx Express Saver

NEW FedEx Extra Hours

Second business day

Third business day

Late drop-off with next business morning delivery to select locations.
Delivery commitment may be later in some areas.

FedEx 1Day Freight*

FedEx 2Day Freight

FedEx 3Day Freight

Next business day

Second business day

Third business day

* Call for Confirmation

5 Packaging

☐ FedEx Envelope*

FedEx Pak*

Other Pkg

Includes FedEx Small Pak, FedEx Large Pak, and FedEx Shrink Pak

Includes FedEx Small Pak, FedEx Large Pak, and FedEx Shrink Pak

Includes FedEx Small Pak, FedEx Large Pak, and FedEx Shrink Pak

* The limit is \$50

6 Special Handling

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HOLD Saturday

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Available only for FedEx Priority Overnight and FedEx 2Day

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Yes

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Dry Ice 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, 60, 65, 70, 75, 80, 85, 90, 95, 100, 105, 110, 115, 120, 125, 130, 135, 140, 145, 150, 155, 160, 165, 170, 175, 180, 185, 190, 195, 200, 205, 210, 215, 220, 225, 230, 235, 240, 245, 250, 255, 260, 265, 270, 275, 280, 285, 290, 295, 300, 305, 310, 315, 320, 325, 330, 335, 340, 345, 350, 355, 360, 365, 370, 375, 380, 385, 390, 395, 400, 405, 410, 415, 420, 425, 430, 435, 440, 445, 450, 455, 460, 465, 470, 475, 480, 485, 490, 495, 500, 505, 510, 515, 520, 525, 530, 535, 540, 545, 550, 555, 560, 565, 570, 575, 580, 585, 590, 595, 600, 605, 610, 615, 620, 625, 630, 635, 640, 645, 650, 655, 660, 665, 670, 675, 680, 685, 690, 695, 700, 705, 710, 715, 720, 725, 730, 735, 740, 745, 750, 755, 760, 765, 770, 775, 780, 785, 790, 795, 800, 805, 810, 815, 820, 825, 830, 835, 840, 845, 850, 855, 860, 865, 870, 875, 880, 885, 890, 895, 900, 905, 910, 915, 920, 925, 930, 935, 940, 945, 950, 955, 960, 965, 970, 975, 980, 985, 990, 995, 1000

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Dry Ice 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, 60, 65, 70, 75, 80, 85, 90, 95, 100, 105, 110, 115, 120, 125, 130, 135, 140, 145, 150, 155, 160, 165, 170, 175, 180, 185, 190, 195, 200, 205, 210, 215, 220, 225, 230, 235, 240, 245, 250, 255, 260, 265, 270, 275, 280, 285, 290, 295, 300, 305, 310, 315, 320, 325, 330, 335, 340, 345, 350, 355, 360, 365, 370, 375, 380, 385, 390, 395, 400, 405, 410, 415, 420, 425, 430, 435, 440, 445, 450, 455, 460, 465, 470, 475, 480, 485, 490, 495, 500, 505, 510, 515, 520, 525, 530, 535, 540, 545, 550, 555, 560, 565, 570, 575, 580, 585, 590, 595, 600, 605, 610, 615, 620, 625, 630, 635, 640, 645, 650, 655, 660, 665, 670, 675, 680, 685, 690, 695, 700, 705, 710, 715, 720, 725, 730, 735, 740, 745, 750, 755, 760, 765, 770, 775, 780, 785, 790, 795, 800, 805, 810, 815, 820, 825, 830, 835, 840, 845, 850, 855, 860, 865, 870, 875, 880, 885, 890, 895, 900, 905, 910, 915, 920, 925, 930, 935, 940, 945, 950, 955, 960, 965, 970, 975, 980, 985, 990, 995, 1000

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Master Index to Studies Submitted Under TSCA 8(e) by 3M Company on October 26, 2004
(Confidential Business Information Redacted)

Title	Substance	CAS Information
Aquatic Toxicity Data Sheet: 48hr <i>Daphnia</i> Magna	1,4-dioxane, heptadecafluoro-1-octanesulfonic acid; linear n-ethyl perfluorooctanesulfonamide; n-ethylperfluorooctanesulfonamidoethyl alcohol; poly(oxy-1,2-ethanediyl), alpha-12- [ethyl]([heptadecafluorooctyl)sulfonylamino]ethyl-, omega-hydroxy-; poly(oxy-1,2-ethanediyl), alpha-12- [ethyl]([nonafluorobutyl)sulfonylamino]ethyl-, omega-hydroxy-; poly(oxy-1,2-ethanediyl), alpha-12- [ethyl]([pentadecafluorohexyl)sulfonylamino]ethyl-, omega-hydroxy-; poly(oxy-1,2-ethanediyl), alpha-12- [ethyl]([tridecafluorohexyl)sulfonylamino]ethyl-, omega-hydroxy-; poly(oxy-1,2-ethanediyl), alpha-12- [ethyl]([undecafluoropentyl)sulfonylamino]ethyl-, omega-hydroxy-; polyethylene glycol; water	1,4-dioxane (123-91-1); heptadecafluoro-1-octanesulfonic acid (1763-23-1); linear n-ethyl perfluorooctanesulfonamide (4151-50-2); n-ethylperfluorooctanesulfonamidoethyl alcohol (1691-99-2); poly(oxy-1,2-ethanediyl), alpha-12-[ethyl]([heptadecafluorooctyl)sulfonylamino]ethyl-, omega-hydroxy- (29117-08-6); poly(oxy-1,2-ethanediyl), alpha-12-[ethyl]([nonafluorobutyl)sulfonylamino]ethyl-, omega-hydroxy- (68298-79-3); poly(oxy-1,2-ethanediyl), alpha-12-[ethyl]([pentadecafluorohexyl)sulfonylamino]ethyl-, omega-hydroxy- (68298-81-7); poly(oxy-1,2-ethanediyl), alpha-12-[ethyl]([tridecafluorohexyl)sulfonylamino]ethyl-, omega-hydroxy- (56372-23-7); poly(oxy-1,2-ethanediyl), alpha-12-[ethyl]([undecafluoropentyl)sulfonylamino]ethyl-, omega-hydroxy- (68298-80-6); polyethylene glycol (25322-68-3); water (7732-18-5)
Multigeneration Daphnid Life Cycle Test	1,4-dioxane, heptadecafluoro-1-octanesulfonic acid; linear n-ethyl perfluorooctanesulfonamide; n-ethylperfluorooctanesulfonamidoethyl alcohol; poly(oxy-1,2-ethanediyl), alpha-12- [ethyl]([heptadecafluorooctyl)sulfonylamino]ethyl-, omega-hydroxy-; poly(oxy-1,2-ethanediyl), alpha-12- [ethyl]([nonafluorobutyl)sulfonylamino]ethyl-, omega-hydroxy-; poly(oxy-1,2-ethanediyl), alpha-12- [ethyl]([pentadecafluorohexyl)sulfonylamino]ethyl-, omega-hydroxy-; poly(oxy-1,2-ethanediyl), alpha-12- [ethyl]([tridecafluorohexyl)sulfonylamino]ethyl-, omega-hydroxy-; poly(oxy-1,2-ethanediyl), alpha-12- [ethyl]([undecafluoropentyl)sulfonylamino]ethyl-, omega-hydroxy-; polyethylene glycol; water	1,4-dioxane (123-91-1); heptadecafluoro-1-octanesulfonic acid (1763-23-1); linear n-ethyl perfluorooctanesulfonamide (4151-50-2); n-ethylperfluorooctanesulfonamidoethyl alcohol (1691-99-2); poly(oxy-1,2-ethanediyl), alpha-12-[ethyl]([heptadecafluorooctyl)sulfonylamino]ethyl-, omega-hydroxy- (29117-08-6); poly(oxy-1,2-ethanediyl), alpha-12-[ethyl]([nonafluorobutyl)sulfonylamino]ethyl-, omega-hydroxy- (68298-79-3); poly(oxy-1,2-ethanediyl), alpha-12-[ethyl]([pentadecafluorohexyl)sulfonylamino]ethyl-, omega-hydroxy- (68298-81-7); poly(oxy-1,2-ethanediyl), alpha-12-[ethyl]([tridecafluorohexyl)sulfonylamino]ethyl-, omega-hydroxy- (56372-23-7); poly(oxy-1,2-ethanediyl), alpha-12-[ethyl]([undecafluoropentyl)sulfonylamino]ethyl-, omega-hydroxy- (68298-80-6); polyethylene glycol (25322-68-3); water (7732-18-5)
Aquatic Invertebrate Testing - Alkylitins LR 8024-1	Alkylitins: dibutyltin laurate and dibutyltin-di(2 ethylhexoate)	Dibutyltin laurate (CAS 77-58-7); Dibutyltin-di(2 ethylhexoate) (CAS 2781-10-4)
Aquatic Invertebrate Testing - Decosheen Material (LR-8052)	Decosheen Ribbon Materials and pigments: Decosheen Blue in Green Ceres Blue ZV; Decosheen Gold Paste Pigment; Decosheen Royal Blue, Solvent Blue	Decosheen Blue in Green (CAS 61814-09-3); Decosheen Royal Blue, Solvent Blue (CAS 61814-09-3); Decosheen Gold Paste Pigment (CAS Number 61814-09-3)
R Scratch Remover (Fathead Minnow)	55-65% Water; 20-30% Stoddard Solvent; 1-5% Sodium Silicate; 1-5% Potassium Hydroxide; 0.1-3% Nonylphenoxypoly(oxyethylene)ethanol	Water (CAS 7732-18-5); Stoddard Solvent (CAS 8052-41-3); Sodium Silicate (CAS 1344-09-8); Potassium Hydroxide (CAS 1310-58-3); Nonylphenoxypoly(oxyethylene)ethanol (CAS 9016-45-9)
S Scratch Remover (Fathead Minnow)	60-70% Water; 20-30% Stoddard Solvent; 1-5% Sodium Silicate; 0.1-3% Turgitol NP-33	Water (CAS 7732-18-5); Stoddard Solvent (CAS 8052-41-3); Sodium Silicate (CAS 1344-09-8); Turgitol NP-33 (CAS 9016-45-9)
Octanol Water Partition Coefficient	N-methylperfluorooctane sulfonamidoethanol	CAS 24448-09-7

CONFIDENTIAL

Master Index to Studies Submitted Under TSCA 8(e) by 3M Company on October 26, 2004
(Confidential Business Information Redacted)

Title	Substance	CAS Information
CoCl ₂ 6H ₂ O as Co ²⁺ + Toxicity to Microtox Reagent	Cobalt (as Co ²⁺ + ion) (CoCl ₂ 6H ₂ O)	CAS 7791-13-1
Activated Sludge Respiration Inhibition Test on CoCl ₂ 6H ₂ O as Co ion	Cobalt (as Co ²⁺ + ion) (CoCl ₂ 6H ₂ O)	CAS 7791-13-1
Acute Toxicity of CoCl ₂ 6H ₂ O as Co ion to <i>Daphnia magna</i> under Static Exposure Conditions	Cobalt (as Co ²⁺ + ion) (CoCl ₂ 6H ₂ O)	CAS 7791-13-1
Acute Toxicity of CoCl ₂ 6H ₂ O as Co ion to Fathead Minnow under Static Exposure Conditions	Cobalt (as Co ²⁺ + ion) (CoCl ₂ 6H ₂ O)	CAS 7791-13-1
Freshwater Algae Growth Inhibition Test	Cobalt (as Co ²⁺ + ion) (CoCl ₂ 6H ₂ O)	CAS 7791-13-1
<i>Daphnia magna</i> 21-Day Chronic Reproduction Study	N-ethylperfluorooctane sulfonamideethanol	CAS 1691-99-2
Plant Growth Effects of []	[]	[]
Final Report (<i>Daphnia</i> and Microtox)	Monomethyl ether of hydroquinone	CAS 150-76-5
Microtox Test Results	2-Ethylhexyl Acrylate; Isooctyl Acrylate Monomer; 2-Methylbutyl acrylate; Methyl Isoamyl acrylate; Isooctyl Acrylate	2-Ethylhexyl Acrylate (CAS 103-11-7); Isooctyl Acrylate Monomer (CAS 29590-42-9) 2-Methylbutyl acrylate (CAS 44914-03-6); Methyl Isoamyl acrylate (CAS 18993-92-1); Isooctyl Acrylate (CAS 29590-42-9)
Phytotoxicity Test Results	[]	[]

Master Index to Studies Submitted Under TSCA 8(e) by 3M Company on October 26, 2004
(Confidential Business Information Redacted)

Title	Substance	CAS Information
Plant Toxicity Comparison, Young Seedling Growth	[REDACTED]	[REDACTED]
<i>Ceriodaphnia dubia</i> Survival and Reproduction exposed to Opequon Creek Water Spiked with BETZ 1110 Polymer (November 4, 1987 sample) for seven days under static renewal conditions	BETZ 1110: Non-3M Product - Chemical composition not provided to 3M by manufacturer	MSDS provided by manufacturer states product is "not hazardous" and not "considered to be a carcinogen"
<i>Ceriodaphnia dubia</i> Survival and Reproduction exposed to Opequon Creek Water Spiked with Betz 1138 Polymer (November 4, 1987 sample) for seven days under static renewal conditions	BETZ 1138: Non-3M Product - Chemical composition not provided to 3M by manufacturer	MSDS provided by manufacturer states product is "not hazardous" and not "considered to be a carcinogen"
Toxicity of 1,6 - Hexanediol Diacrylate to <i>Daphnia magna</i>	1,6 Hexanediol diacrylate	CAS 13046-33-4
<i>Daphnia magna</i> Chronic Bioassay Under Static Renewal Conditions	Methyl isoamyl acrylate	CAS 18993-92-1
Estimating the Chronic Toxicity of Nalclear 7177 to <i>Ceriodaphnia</i> Survival and Reproduction Using Short-Term Tests	Nalclear 7177 wastewater treatment acrylamide/acrylate polymer - Chemical composition not provided to 3M by manufacturer	CAS Information not provided to 3M by manufacturer
Acute Toxicity of Isooctyl Acrylate to <i>Daphnia magna</i>	Isooctyl Acrylate Monomer	CAS 29590-42-9
Static Acute Toxicity of [REDACTED] to the Daphnid, <i>Daphnia magna</i>	Tolyltriazole	CAS 29385-43-1
Static Acute Toxicity of [REDACTED] to the Alga, <i>Selenastrum capricornutum</i>	Tolyltriazole	CAS 29385-43-1
Static Acute Toxicity of [REDACTED] to the Daphnid, <i>Daphnia magna</i>	[REDACTED]	[REDACTED]
Static Acute Toxicity of [REDACTED] to the Fathead Minnow, <i>Pimephales promelas</i>	[REDACTED]	[REDACTED]
Static Acute Toxicity of [REDACTED] to the Daphnid, <i>Daphnia magna</i>	water; propylene-tetrafluoroethylene polymer; tert-butyl alcohol	water (7732-18-5); propylene-tetrafluoroethylene polymer (27029-05-6); tert-butyl alcohol (75-65-0)

Master Index to Studies Submitted Under TSCA 8(e) by 3M Company on October 26, 2004
(Confidential Business Information Redacted)

Title	Substance	CAS Information
Isocetyl acrylate: Fish, Acute Toxicity Test	Isocetyl Acrylate Monomer	CAS 29590-42-9
Isocetyl Acrylate: <i>Daphnia</i> sp. Acute Immobilization Test	Isocetyl Acrylate Monomer	CAS 29590-42-9
Isocetyl Acrylate: Alga, Growth Inhibition Test	Isocetyl Acrylate Monomer	CAS 29590-42-9
Isocetyl Acrylate: <i>Daphnia</i> sp. Reproduction Test	Isocetyl Acrylate Monomer	CAS 29590-42-9
Acute Toxicity of [] to the mysid, <i>Mysidopsis bahia</i>	[]	[]
Final Report (Microtox)	[]	[]
Determination of the Partition Coefficient (N-Octanol/Water) of T-5896 by High Performance Liquid Chromatography (HPLC)	N-methyl perfluorooctane sulfonamido ethanol; N-methyl perfluorooctane sulfonamidoethyl acrylate	N-methyl perfluorooctane sulfonamido ethanol (CAS 25268-77-3); N-methyl perfluorooctane sulfonamidoethyl acrylate (CAS 24448-09-7)
OECD Activated Sludge Respiration Inhibition Test Results	N-Dodecyltrimethylammonium chloride	CAS = 112-00-5
Final Report (Fish Acute Toxicity)	Mirlatine CB (30% Cocamidopropyl betaine = Amides, coco, N-(3-(dimethylamino)propyl), alkylation products with chloroacetic acid, sodium salts, 70% Water and Inerts); Mirlatine COB (30% Coco/Oleamidopropyl Betaine = 1-Propanaminium, 3-amino-N-(carboxymethyl)-N,N-dimethyl-, N-coco acyl derivs., inner salt)	Cocamidopropyl betaine (CAS 70851-07-9); Coco/Oleamidopropyl Betaine (CAS 61789-40-0)
A Flow-Through Life-Cycle Toxicity Test With the Saltwater Mysid (<i>Mysidopsis bahia</i>)	Perfluorooctane sulfonate	CAS 1763-23-1
Lithium: Alga, Acute toxicity Tests	Lithium Chloride	CAS 7447-41-8
An Early Life-Stage Toxicity Test With the Fathead Minnow (<i>Pimephales promelas</i>)	Perfluorooctane sulfonate	CAS 1763-23-1
Lithium: Fish, Acute toxicity Tests	Lithium Chloride	CAS 7447-41-8
Lithium: <i>Daphnia</i> , Acute toxicity Tests	Lithium Chloride	CAS 7447-41-8
Summary of Toxicity Testing on OSCI and OSF	Octane sulfonyl chloride and Octane sulfonyl fluoride	Octane sulfonyl fluoride (CAS 7795-95-1), Octane sulfonyl chloride (CAS 4063-63-5)
Toxicity to Microtox Test	Lauryldimethylamineoxide	CAS 1643-20-5

Master Index to Studies Submitted Under TSCA 8(e) by 3M Company on October 26, 2004
(Confidential Business Information Redacted)

Title	Substance Identification	CAS Information
Ecotoxicological Testing of CoCl ₂ .6H ₂ O as Co ²⁺ Ion (Seed Germination and Root Elongation)	Cobalt (as Co ²⁺ ion) (CoCl ₂ .6H ₂ O)	CAS 7791-13-1

CERTIFICATION OF GOOD LABORATORY PRACTICE COMPLIANCE

To the best of my knowledge, this study was conducted in accordance with OECD Good Laboratory Practice Standards (OECD Council Decision C(81)30, Annex 2: OECD Principles of Good Laboratory Practice 1981).

Study Director: Dinesh Vaishnav Date: 7-13-92
Dinesh Vaishnav
ASCI Corporation/ASCI-Duluth
Environmental Testing Division

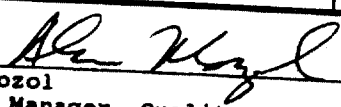
Based on the signatures of the Study Director and the Quality Assurance Auditor, this study, to the best of our knowledge, was conducted in accordance with OECD Good Laboratory Practice Standards (OECD Council Decision C(81)30, Annex 2: OECD Principles of Good Laboratory Practice 1981).

Sponsor: Richard P. ... Date: 7/14/92
Submitter: Annun A. Beach Date: 7/14/92

STATEMENT OF QUALITY ASSURANCE

The study data were reviewed by the ASCI-Duluth Environmental Testing Division Quality Assurance Unit to assure that standard operating procedures and guidelines used to conduct this study were followed, and this report is an accurate reflection of the raw data. The types of audits performed are listed in the following table.

Type of Audit for ASCI Study ID# 5030-003-06	Audit Date	Date Reported to Study Director and Management
Study Plan	11-14-1991	11-17-1991
In-Life Phase	03-24-1992	03-24-1992
Raw Data and Draft Report	06-16-1992	06-16-1992
Final Report	07-12-1992	07-14-1992


Alan Mozol
Acting Manager, Quality Assurance Unit

Date: 7/12/92

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STUDY SUMMARY TABLE

Study Title	Isooctyl Acrylate: Alga, Growth Inhibition Test
Data Standard	OECD Guideline 201, and Good Laboratory Practice standards as promulgated under the OECD Council Decision C(81)30, Annex 2: OECD Principles of Good Laboratory Practice.
Sponsor	Rich Purdy, 3M Environmental Laboratory, Building 2-3E-09, 935 Bush Avenue, St. Paul, MN 55106; Tel No. (612) 778-5379.
Sponsor's Representative	Susan A. Beach, 3M Environmental Laboratory, Building 2-3E-09, 935 Bush Avenue, St. Paul, MN 55106; Tel No. (612) 778-7452.
Testing Facility	ASCI Corporation/ASCI-Duluth Environmental Testing Division, 112 East Second Street, Duluth, MN 55805; Tel. No. (218) 722-4040.
Study Director	Dinesh Vaishnav
Acting QAU Manager	Alan Mozol
Testing Facility Director	Donald Mount
Study Initiation Date	March 20, 1992.
Test Dates	March 20-24, 1992.
Test Substance	Isooctyl acrylate (CAS No. 29590-42-9, [REDACTED] Lot 3290), 99.7% acrylate (as determined by Sponsor [NB# [REDACTED]] liquid.
Test Organism	<i>Selenastrum capricornutum</i> (ATCC 22662).

Test Description	<p>(1) Control and test exposures (each 200 ml volume) were prepared using 500-ml separatory funnels with ground glass stoppers, (2) the funnels were inoculated with the algal cells and incubated for 96 h on a rotary shaker (operating at 150 rpm) at 21.5-24.3°C under fluorescent lamps providing continuous and uniform illumination of 86-91 $\mu\text{E}/\text{m}^2\text{s}$, (3) the algal cell concentrations, the algal medium chemistry parameters and the test substance concentrations were determined at appropriate time intervals, (4) the algal cell concentration data were used to derive the average specific growth rates, and (5) the latter were used to calculate the 96-h ERC50 and 96-h NOEC values, based on mean measured test substance concentrations.</p>
Test Results	<p>Based on the mean of the measured concentrations which were not corrected for the daily algal medium spike recovery, isooctyl acrylate 96-h ERC50 and 96-h NOEC for a green alga (<i>S. capricornutum</i>) were 1.74 mg/L and 1.30 mg/L, respectively.</p> <p>Based on the mean of the measured concentrations which were corrected for the daily algal medium spike recovery, isooctyl acrylate 96-h ERC50 and 96-h NOEC for a green alga (<i>S. capricornutum</i>) were 2.13 mg/L and 1.70 mg/L, respectively.</p>
Location of Raw Data and Final Report	<p>ASCI Corporation/ASCI-Duluth Environmental Testing Division, 112 East Second Street, Duluth, MN 55805; Tel. No. (218) 722-4040.</p>

1.0 INTRODUCTION

The test substance, isooctyl acrylate, is an ester primarily made from isooctanol and acrylic acid. It has negligible solubility in freshwater and its effect on the growth of a unicellular green algal species is not known. The purpose of the present study was to determine if possible, the 96-h ERC50 and 96-h NOEC (no observed effect concentration) of the test substance for an alga (*Selenastrum capricornutum*, ATCC 22662). The study was conducted according to the ASCI study plan.

The ERC50 is that test substance estimated concentration, which in a specified time should cause a 50% reduction in the average specific growth rate (μ) of the test organism when compared to the control value. The 96-h NOEC is the highest test substance concentration tested at which no significant reduction in average specific growth rate (μ) of the test organism occurs in 96 h when compared to the control value.

2.0 TEST METHODS

2.1 Test Substance. The test substance, isooctyl acrylate (CAS No. 29590-42-9, [] Lot 3290), was received at ASCI on October 3,

Sponsor: 3M Company
Sponsor Study RM 17714

1991 in one amber glass bottle placed in a sealed metal container. The test substance was stored at room temperature as received. According to a material safety data sheet and a written communication provided by the Sponsor (Appendix A), (1) the test substance was a clear, colorless, mobile liquid with acrylate odor, (2) the test substance has negligible water solubility and 1 mm Hg vapor pressure at 50°C, (3) the test substance is 99.75% acrylate as determined by [] (4) the test substance is stable and its biodegradation ranged from 59%-85% in five days, and (5) the test substance concentration in deionized water can be analyzed by a GC method. The Sponsor also has information that, based on the chemical structure, there will be essentially no dissociation of the test substance at environmental pH levels. The Sponsor suspects the test substance may have glass surface activity.

2.2 General Test Conditions. All glassware, pipets, tubings, algal medium and reagents used in this test were autoclaved for 15 minutes at 121°C (15 psi pressure). When necessary, aseptic techniques were also used to prevent microbial contamination of the subject materials.

2.3 Test Substance Solutions. To prepare the test substance stock solution, 24 h before the test initiation, 20 μ l of the test substance were added to 2 L of the algal medium contained in a 2-L separatory funnel. To equilibrate the test substance concentration, the funnel was closed and incubated for 24 h on a rotary shaker (operating at 150 rpm) at 21.5-24.3°C under fluorescent lamps providing continuous and uniform illumination of 86-91 μ E/m²s. The pH of the stock solution was 6.43 and was not adjusted.

For use in this test, the test substance nominal concentrations of 0.0 (inoculum control; less than the method detection limit of 0.04 mg/L), 0.6, 1.2, 2.4, 4.7 and 9.4 mg/L were prepared from the equilibrated, analytically measured stock solution. The same nominal concentrations, when corrected for the algal medium spike recovery of 89% at test initiation, were calculated to be 0.0 (inoculum control; less than the method detection limit of 0.04 mg/L), 0.7, 1.3, 2.7, 5.3, and 10.6 mg/L. To achieve the nominal concentrations, the required volumes of the stock solution and the algal medium were directly mixed into the test and control separatory funnels.

2.4 Algal Medium. The following algal medium (Nichols 1973) was used:

Chemical	g/L Stock solution	mg/L Algal medium
Macronutrients (prepared seven individual stock solutions)		
$\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$	36.76	36.76
$\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$	36.97	36.97
NaHCO_3	12.60	12.60
K_2HPO_4	8.71	8.71
NaNO_3	85.01	85.01
Na_2EDTA	4.36	4.36
$\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$	3.15	6.30
Micronutrients (prepared one stock solution)		
$\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$	0.01	0.01
$\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$	0.022	0.22
$\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$	0.18	0.18
$\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$	0.006	0.006
H_3BO_3	1.0	1.0

The stock solutions of the above chemicals were prepared in deionized water and stored in plastic bottles in dark at 2-4°C. All stock solutions were brought to a room temperature and mixed well before using them.

To prepare an algal medium for use during this test, 1 ml each of the six macronutrient stock solutions and 1 ml of the micronutrient stock solution were added to each 1 L of deionized water. The macronutrient stock solution of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ was added at a rate of

2 ml/L of deionized water. The macronutrient stock solution of Na₂ EDTA was not used when the medium was prepared for conducting the toxicity test. The medium was then dispensed in Erlenmeyer flasks and autoclaved. The medium was aerated for 15 h on a rotary shaker (operating at 150 rpm) at 21.5-24.3°C before using it.

The chemical analysis of deionized water is performed annually and the most recent analyses are provided in Appendix A.

2.5 Algal Inoculum. To prepare an algal inoculum, 200 ml of the aerated algal medium were dispensed in a 500-ml separatory funnel and aseptically inoculated with 1 ml of the algal stock culture maintained at ASCI. The funnel was then closed and incubated on a rotary shaker (operating at 150 rpm) at 21.5-24.3°C under fluorescent lamps providing continuous and uniform illumination of 86-91 $\mu\text{E}/\text{m}^2/\text{s}$. Upon development of the visible algal biomass, it was serially subcultured twice into the fresh algal medium. The final subculture was centrifuged (2°C, 995g force), washed and diluted using an algal medium to provide approximately 1.5×10^4 cells/ml in the test. The final subculture was 3-day old.

2.6 Algal Growth Inhibition Test Setup. The test was conducted using 18, 500-ml separatory funnels (final volume 201 ml/funnel);

15 with the test substance (test funnels) and three with inoculum only (inoculum control funnels). During the incubation, each funnel was closed with a glass stopper and samples for the algal cell counts and test substance analysis were collected through the stem of the funnel while keeping the closure slightly open. To setup the test:

- (1) Dispensed the required volumes of the aerated algal medium in triplicate test and inoculum control funnels;
- (2) While keeping the closure slightly open, dispensed the required volumes of the equilibrated test substance stock solution to triplicate test and inoculum control funnels (combined volume 200 ml/funnel), and mixed the content of each funnel;
- (3) Aseptically inoculated each funnel with 1 ml of the algal inoculum to obtain approximately 1.5×10^4 cells/ml of algal medium (final volume 201 ml/funnel), closed funnels; and
- (4) Incubated them for 96 h on a rotary shaker (operating at 150 rpm) at 21.5-24.3°C under cool white fluorescent lamps providing 86-91 $\mu\text{E/m}^2\text{s}$ continuous and uniform illumination.

2.7 Determination of Algal Growth. At test initiation (0 h) and thereafter at 24 h, 48 h, 72 h and 96 h (test termination), algal cell concentrations in test and inoculum control funnels were determined by a direct microscopic count technique (APHA 1980). At test initiation, the counts were made using one replicate each of low, middle and high test substance concentration, and inoculum control. All other counts during the test were made using individual replicate samples. The replicate counts were averaged and used in data analysis.

2.8 Determination of Other Test Parameters. During the test, incubation temperature, shaker function and illumination were determined twice daily. The Ph of the content of one replicate funnel containing low, middle and high test substance concentration, and inoculum control was determined at test initiation (0 h) and termination (96 h).

2.9 Test Substance Analysis. The test substance concentrations in individual and composite samples were analyzed according to the following schedule:

Type of Sample	Frequency of Sampling	Total Number of Samples Analyzed
Stock solution (same as high concn)	Did not sample separately.	Did not analyze separately.
Test and control	0 h	6 composite samples
Test and control	24 h	6 composite samples
Test and control	48 h	6 composite samples
Test and control	72 h	6 composite samples
Test and control	96 h	18 individual samples

Each sample was collected directly from the stem of the funnel into a 60-ml brown glass sample bottle. The bottles were graduated to 20 ml, 40 ml and 60 ml volumes. The composite samples were prepared by combining 20 ml replicate samples. The samples were extracted and analyzed using the procedures described in the analytical method validation report (Appendix B). If required, a sample concentration was performed under a nitrogen stream before analyzing the sample for the test substance.

2.10 Treatment of Results. Natural log of the mean algal cell concentration and incubation time were correlated for each treatment, and the slope of the regression line was taken as an average specific growth rate (μ). The data used in each correlation were of the exponentially growing algal cells as determined from

the plot of the log of the mean algal cell concentration and incubation time.

A percentage reduction in the average specific growth rate at each test substance concentration was calculated in comparison to the control value. These data were then used to calculate the 96-h ERC50 and 96-h NOEC using trimmed Spearman-Kärber method (Hamilton et. al. 1977) and the TOXSTAT, Version 3.1 (University of Wyoming, Laramie, Wyoming 1989) software, respectively. The ERC50 and NOEC values were computed and reported on the basis of the test substance mean measured concentrations both uncorrected and corrected for the daily algal medium spike recoveries

3.0 RESULTS

The algal average specific growth rates in 96 h ranged from 0.01 cells ml⁻¹ h⁻¹ in the highest three test exposures to 0.03 cells ml⁻¹ h⁻¹ in the control exposure (Table 1). Similarly, inhibitions of the growth rates ranged from 0% in the control exposure to 67% in the highest three test exposures (Table 1).

Based on the mean of the measured concentrations which were not corrected for the daily algal medium spike recovery, the test substance 96-h ErC50 was 1.74 mg/L and the 96-h NOEC was 1.39 mg/L (Table 2). Similarly, based on the mean of the measured concentrations which were corrected for the daily algal medium spike recovery, the test substance 96-h ErC50 was 2.13 mg/L and the 96-h NOEC was 1.70 mg/L (Table 2).

The volumes of the algal medium and the test substance stock solution mixed to achieve the nominal concentrations are given in Table 3. All nominal concentrations were calculated based on the analytically measured stock solution and a dilution factor of 2.0. The nominal concentrations were computed in two different manners; in one case the stock concentration was not corrected, and in the other case it was corrected, for the algal medium spike recovery of 89% at test initiation.

The data for standard (deionized water) and test (algal medium) matrix spike recoveries are presented in Table 4. The mean spike recovery from deionized water was $81 \pm 4.8\%$ and from algal medium $82 \pm 8.2\%$ (Table 4).

The test substance measured concentrations and recoveries are presented in Tables 5 and 6. In the one case, the test substance daily measured concentrations were not corrected for the algal medium spike recovery before calculating the recoveries from the test exposures (Table 5). In the other case, the test substance daily measured concentrations were corrected for the algal medium spike recovery for that particular day before calculating the recoveries from the test exposures (Table 6). For example, the test substance final measured concentrations were corrected for 89% (Table 6), as algal medium spike recovery for 96 h was 89% (Table 4).

The test substance uncorrected mean recoveries were between 81% and 153% with 9.4 mg/L and 0.6 mg/L test substance nominal concentrations, respectively (Table 5). The test substance corrected mean recoveries were between 89% and 163% with 10.6 mg/L and 0.7 mg/L test substance nominal concentrations, respectively (Table 6).

In 72 h, the mean algal cell concentration in the control exposure increased by a factor of about 8 (Table 7) and in 96 h by a factor of 11. The desired factor for an increase in 72 h is 16 when a test is performed using, for example, 250-ml Erlenmeyer flasks each containing 50 ml of algal medium. The smaller increases in the

present test may have been due to the type of exposure vessels (500-ml closed separatory funnels each containing 200 ml of algal medium) used. One limitation of the exposure vessels used was that they did not allow for any air exchange or the injection of ambient carbon dioxide, which both are essential for the algal cells to propagate. The use of the separatory funnels, however, was also necessary to minimize the loss of the test substance due to evaporation, especially because the exposure vessels were incubated on a rotary shaker operating at 150 rpm.

At test initiation, the exposure medium pH values were between 6.43 and 6.75, and at test termination they were between 6.95 and 9.77 (Table 8). During the test, the incubation was at 150 rpm, the temperature was between 21.5°C and 23.1°C, and light intensity was between 400 ft-c or 86 $\mu\text{E}/\text{m}^2\text{s}$ and 425 ft-c or 91 $\mu\text{E}/\text{m}^2\text{s}$ (Table 9).

From the quality assurance standpoint, this test is acceptable because it complies with both acceptance criteria, especially when the explanation for a smaller increase in algal biomass concentration is considered (Table 10).

4.0 CONCLUSIONS

Based on the mean of the measured concentrations which were not corrected for the daily algal medium spike recoveries, isooctyl acrylate 96-h ErC50 and 96-h NOEC for *S. capricornutum* (ATCC 22662) were 1.74 mg/L and 1.39 mg/L, respectively. Similarly, based on the mean of the measured concentrations which were corrected for the daily algal medium spike recoveries, the 96-h ErC50 and the 96-h NOEC were 2.13 mg/L and 1.70 mg/L, respectively.

5.0 DEVIATIONS FROM APPROVED ASCI STUDY PLAN

The deviations which occurred while conducting this study were:

- (1) The test substance stock solution was prepared in 2 L algal medium contained in a 2-L separatory funnel instead of 1 L algal medium contained in a 2-L separatory funnel. The pH of the stock solution was 6.43 instead of 8. To equilibrate the test substance concentration, the funnel was closed and incubated for 24 h at 21.5-24.3°C, instead of 15-20 h at $23 \pm 2^\circ \text{C}$, under cool white fluorescent lamps providing 86-91 $\mu\text{E}/\text{m}^2/\text{s}$ continuous and uniform illumination.


- (2) The algal medium for use in the test was equilibrated with air for 15 h instead of 24 h before using it.
- (3) To setup the test, dispensed the required volumes of the aerated algal medium in triplicate test and inoculum control funnels;
- (4) While keeping the closure slightly open, dispensed the required volumes of the equilibrated test substance stock solution to triplicate test and inoculum control funnels (combined volume 200 ml/funnel), and mixed the content of each funnel;
- (5) Aseptically inoculated each funnel with 1 ml of the algal inoculum to obtain approximately 1.5×10^4 cells/ml of algal medium (final volume 201 ml/funnel), closed funnels;
- (6) Incubated them for 96 h on a rotary shaker (operating at 150 rpm) at 21.5-24.3°C under cool white fluorescent lamps providing 86-91 $\mu\text{E}/\text{m}^2\text{s}$ continuous and uniform illumination; and
- (7) Instead of 24-h, 48-h, 72-h and 96-h ErC_{50} values, only 96-h ErC_{50} was calculated because the test substance effect was measured in terms of the average specific growth rate (μ) at each treatment. The average specific growth rate (μ) was the calculated mean growth rate for

the entire test period of 96 h, and it was the slope of the linear regression of the natural log of the mean algal cell concentrations and incubation times. This approach to the data analysis did not allow calculating individual growth rates at 24, 48, 72 and 96 h.

To the best of our current scientific knowledge and understanding, these deviations should have no effect on the results presented in this report.

6.0 REPORT SIGNATURE

Study Director:



Date: 7-13-92

Dinesh Vaishnav
ASCI Corporation/ASCI-Duluth
Environmental Testing Division

7.0 REFERENCES

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8.0 PERSONNEL INVOLVED IN STUDY AND THEIR RESPONSIBILITIES

Personnel	Responsibility
Dinesh Vaishnav	Study Director
Minren Xu	Analytical chemistry
Billie Samson	Laboratory assistance
Dave Nessa	Laboratory assistance
Romesh Lakhan	Glassware preparation
Alan Mozol	QA
Nancy Jordan	Archivist

Table 1. Isooctyl acrylate (test substance): Average specific growth rate (μ = cells $\text{ml}^{-1} \text{h}^{-1}$) of algal (*S. capricornutum*) cells and other pertinent statistics

Test substance mean measured concn (mg/L) ^a		Data used in regression	r ²	Rate \pm SD/h and % rate inhibition
Uncorrected	Corrected			
0.0 (inoculum control)	<MDL ^b	5 (0-96 h)	0.935	0.03 \pm 0.004 (0)
0.92	1.13	5 (0-96 h)	0.836	0.02 \pm 0.005 (33%)
1.39	1.70	4 (24-96 h)	0.953	0.02 \pm 0.002 (33%)
2.18	2.66	3 (24, 48, 96 h)	0.973	0.01 \pm 0.001 (67%)
4.28	5.22	4 (24-96 h)	0.883	0.01 \pm 0.003 (67%)
7.61	9.39	4 (24-96 h)	0.963	0.01 \pm 0.003 (67%)

^aIn one case the measured concentrations were not corrected, and in the other case they were corrected, for the daily algal medium spike recoveries.

^bMethod detection limit (MDL) was 0.04 mg/L test substance.

Table 2. Isooctyl acrylate (test substance): 96-h ErC50 and
 96-h NOEC values for algal (*S. capricornutum*) cell
 average specific growth rate (μ)

Data used for calculation	96-h ErC50 (mg/L) and 95% confidence limits*	96-h NOEC (mg/L)
Mean of the measured concentrations which were not corrected for the daily algal medium spike recoveries.	1.74 (1.59-1.90)	1.39
Mean of the measured concentrations which were corrected for the daily algal medium spike recoveries.	2.13 (1.95-2.32)	1.70

*Limits are in parenthesis.

Table 3. Isooctyl acrylate (test substance): Volumes of algal medium and test substance stock solution mixed to achieve test substance nominal concentrations

Algal medium (ml)	Test substance stock solution (ml)	Total volume (ml)	Test substance nominal concn (mg/L)*	
			Uncorrected	Corrected
200	0.0	200	0.0 (inoculum control)	0.0 (inoculum control)
187.5	12.5	200	0.6	0.7
175	25	200	1.2	1.3
150	50	200	2.4	2.6
100	100	200	4.7	5.3
0.0	200	200	9.4	10.6

*Nominal concentration was based on the analytically measured stock solution and a dilution factor of 2.0. In one case the stock concentration was not corrected, and in the other case it was corrected, for the algal medium spike recovery of 89% at test initiation.

Table 4. Isooctyl acrylate (test substance): Spike recoveries

Matrix	Time of analysis	Test substance concn (mg/L)		% Recovery ^a
		Target	Measured	
Matrix-- Deionized water				
Method blank	0 h	0.0	<MDL ^b	NC ^c
	24 h	0.0	<MDL	NC
	72 h	0.0	<MDL	NC
	96 h	0.0	<MDL	NC
Spike solution	0 h	0.22	0.19	87
	24 h	0.50	0.39	79
	72 h	0.50	0.38	76
	96 h	0.30	0.25	83
Mean spike recovery 81 ± 4.8%				
Matrix-- Algal medium				
Method blank	0 h	0.0	<MDL	NC
	24 h	0.0	<MDL	NC
	72 h	0.0	<MDL	NC
	96 h	0.0	<MDL	NC
Spike solution	0 h	0.50	0.45	89
	24 h	0.50	0.36	73
	72 h	0.50	0.39	77
	96 h	0.50	0.45	89
Mean spike recovery 82 ± 6.2%				

^aSpike recoveries were computed using Minitab[®] statistical software and rounded-off before reporting in this table.

^bMethod detection limit (MDL) was 0.04 mg/L test substance.

^cNC = not calculated.

Table 5. Isooctyl acrylate (test substance): Uncorrected nominal and measured concentrations^a

Test substance nominal concn (mg/L)	Rep	Test substance measured concn (mg/L)					Mean \pm SD	% Recovery ^b
		0 h	24 h	48 h	72 h	96 h		
0.0 (inoculum control)	A	- ^c	-	-	-	<MDL ^d		
	B	-	-	-	-	<MDL		
	C	-	-	-	-	<MDL		
	All ^e	<MDL	<MDL	<MDL	<MDL	<MDL		
0.6	A	-	-	-	-	1.16	NC ^f	NC
	B	-	-	-	-	0.59		
	C	-	-	-	-	0.73		
	All	0.96	0.95	1.08	0.97	-	0.92 \pm 0.196	153
1.2	A	-	-	-	-	0.94		
	B	-	-	-	-	1.37		
	C	-	-	-	-	1.23		
	All	1.78	1.18	1.60	1.66	-	1.39 \pm 0.301	116
2.4	A	-	-	-	-	2.05		
	B	-	-	-	-	2.17		
	C	-	-	-	-	2.16		
	All	2.07	1.88	2.09	2.81	-	2.18 \pm 0.295	91
Continued on the next page.								

Table 5 (continued)

Test substance nominal concn (mg/L)	Rep	Test substance measured concn (mg/L)					Mean \pm SD	% Recovery ^b
		0 h	24 h	48 h	72 h	96 h		
4.7	A	-	-	-	-	4.34		
	B	-	-	-	-	4.76		
	C	-	-	-	-	3.92		
	All	3.89	3.73	4.72	4.61	-	4.28 \pm 0.432	91
9.4	A	-	-	-	-	5.98		
	B	-	-	-	-	-		
	C	-	-	-	-	7.85		
	All	9.42	8.19	7.06	7.18	-	7.61 \pm 1.168	81

^aNominal and measured concentrations were not corrected for the daily algal medium spike recovery.

^bPercentage recovery = (mean measured concentration/nominal concentration) \times 100.

- = Not determined.

^cMethod detection limit (MDL) was 0.04 mg/L test substance.

^dAll = composite sample prepared from replicate samples.

^eNC = not calculated.

Table 6. Isooctyl acrylate (test substance): Corrected nominal and measured concentrations^a

Test substance nominal concn (mg/L)	Rep	Test substance measured concn (mg/L)					Mean \pm SD	% Recovery ^b
		0 h	24 h	48 h	72 h	96 h		
0.0 (inoculum control)	A	-	-	-	-	<MDL ^c		
	B	-	-	-	-	<MDL		
	C	-	-	-	-	<MDL		
	All ^d	<MDL	<MDL	<MDL	<MDL	<MDL		
0.7	A	-	-	-	-	NC ^e		NC
	B	-	-	-	-	1.30		
	C	-	-	-	-	0.67		
	All	1.08	1.30	1.47	1.26	0.82		
1.3	A	-	-	-	-	1.13 \pm 0.291		163
	B	-	-	-	-	1.05		
	C	-	-	-	-	1.53		
	All	2.00	1.61	2.19	2.15	1.38		
2.6	A	-	-	-	-	1.70 \pm 0.427		132
	B	-	-	-	-	2.31		
	C	-	-	-	-	2.44		
	All	2.32	2.58	2.87	3.65	2.42		
Continued on the next page.							2.66 \pm 0.476	103

Table 6 (continued)

Test substance nominal concn (mg/L)	Rep	Test substance measured concn (mg/L)					Mean \pm SD	% Recovery ^b
		0 h	24 h	48 h	72 h	96 h		
5.3	A	-	-	-	-	4.88		
	B	-	-	-	-	5.35		
	C	-	-	-	-	4.41		
	All	4.37	5.11	6.47	5.99	-	5.22 \pm 0.783	99
10.6	A	-	-	-	-	6.72		
	B	-	-	-	-	-		
	C	-	-	-	-	8.12		
	All	10.58	11.22	9.67	9.32	-	9.39 \pm 1.570	89

^aNominal and measured concentrations were corrected for the daily algal medium spike recovery (Table 4).

^bPercentage recovery = (mean measured concentration/nominal concentration) X 100.

^c- = Not determined.

^dMethod detection limit (MDL) was 0.04 mg/L test substance.

^eAll = composite sample prepared from replicate samples.

^fNC = not calculated.

Table 7. Isooctyl acrylate (test substance); Algal
(*S. capricornutum*) cell concentrations

Test substance nominal concn (mg/L) ^a		R e p	Algal cell concentrations (per milliliter) at various incubation times				
Uncorrected	Corrected		0 h	24 h	48 h	72 h	96 h
0.0 (inoculum control)	0.0 (inoculum control)	A	3.2E 4 ^b	5.2E 4	2.1E 5	2.5E 5	3.4E 5
		B	ND ^c	6.6E 4	2.1E 5	2.4E 5	3.5E 5
		C	ND	8.0E 4	2.1E 5	2.7E 5	4.6E 5
Mean ^d			3.4E 4	6.6E 4	2.1E 5	2.5E 5	3.8E 5
± SD ^d			0.06	1.40	0.03	0.14	0.70
0.6	0.7	A	ND	4.4E 4	3.6E 4	6.0E 4	1.3E 5
		B	ND	4.4E 4	4.6E 4	1.1E 5	2.7E 5
		C	2.8E 4	3.8E 4	3.4E 4	1.1E 5	1.6E 5
Mean			3.4E 4	4.2E 4	3.9E 4	9.2E 4	1.9E 5
± SD			0.06	0.35	0.64	2.77	0.73
1.2	1.3	A	ND	3.0E 4	2.4E 4	5.6E 4	8.0E 4
		B	ND	2.2E 4	3.4E 4	3.6E 4	5.4E 4
		C	ND	2.0E 4	2.4E 4	4.4E 4	6.2E 4
Mean			NC ^e	2.4E 4	2.7E 4	4.5E 4	6.5E 4
± SD			NC	0.53	0.58	1.01	1.33
2.4	2.6	A	3.8E 4	2.8E 4	3.4E 4	2.8E 4	4.0E 4
		B	ND	3.0E 4	3.4E 4	2.6E 4	4.4E 4
		C	ND	3.2E 4	2.8E 4	3.4E 4	4.6E 4
Mean			3.4E 4	3.0E 4	3.2E 4	2.9E 4	4.3E 4
± SD			0.06	0.20	0.35	0.42	0.31
Continued on the next page.							

Table 7 (continued)

Test substance nominal concn (mg/L) ^a		R e p	Algal cell concentrations (per milliliter) at various incubation times				
Uncorrected	Corrected		0 h	24 h	48 h	72 h	96 h
4.7	5.3	A	ND	1.2E 4	1.6E 4	2.8E 4	3.2E 4
		B	ND	2.2E 4	2.0E 4	2.2E 4	4.6E 4
		C	ND	1.8E 4	1.6E 4	2.4E 4	3.8E 4
Mean			NC	1.7E 4	1.7E 4	2.5E 4	3.9E 4
± SD			NC	0.50	0.23	0.31	0.70
9.4	10.6	A	ND	1.6E 4	3.0E 4	5.4E 4	4.4E 4
		B	ND	2.4E 4	3.0E 4	2.6E 4	4.8E 4
		C	4.0E 4	2.2E 4	2.4E 4	1.6E 4	1.8E 4
Mean			3.4E 4	2.1E 4	2.8E 4	3.2E 4	3.7E 4
± SD			0.06	0.42	0.35	1.97	1.63

^aNominal concentration was based on the analytically measured stock solution and a dilution factor of 2.0. In one case the stock concentration was not corrected, and in the other case it was corrected, for the algal medium spike recovery of 89% at test initiation.

^bIn all cases, the number after E is an exponential power to the base 10.

^cND = not determined.

^dAt test initiation, cell concentrations were measured in inoculum control and low, middle and high test exposures, and mean and SD values were calculated based on the four, measured concentrations.

^eNC = not calculated.

Table 8. Isooctyl acrylate (test substance): pH of selected exposures

Test substance nominal concn (mg/L)*		pH at test initiation	pH at test termination
Uncorrected	Corrected		
0.0 (inoculum control)	0.0 (inoculum control)	6.72	9.77
0.6	0.7	6.75	7.74
2.4	2.6	6.65	7.22
9.4	10.6	6.43	6.95
Ranges		6.43-6.75	6.95-9.77

*Nominal concentration was based on the analytically measured stock solution and a dilution factor of 2.0. In one case the stock concentration was not corrected, and in the other case it was corrected, for the algal medium spike recovery of 89% at test initiation.

Table 9. Isooctyl acrylate (test substance): Incubation conditions in growth chamber

Incubation time	Shaker rpm	Temperature (°C) (at two points)		Light Intensity (ft-c) (at two points)	
		A.M. hours	P.M. hours	A.M. hours	P.M. hours
0 h	150	Not determined	24.3, 23.5	Not determined	425, 400
24 h	150	21.8, 21.9	21.5, 21.5	400, 400	400, 400
48 h	150	21.9, 22.0	21.8, 21.9	400, 400	400, 400
72 h	150	22.4, 22.4	22.1, 22.3	400, 400	400, 400
96 h	150	23.1, 23.0	Test ended	400, 400	Test ended
Ranges	150	21.5-23.1		400-425 (86-91 $\mu\text{E}/\text{m}^2\text{s}$)	

Table 10. Isooctyl acrylate (test substance): QA criteria and test acceptability

Criterion	Results
Mean algal biomass in inoculum control funnels must increase by a factor of 16 within 72 h.	The increase was by a factor of 8, possibly because the type of exposure vessels (500-ml closed separatory funnels each containing 200 ml of algal medium) used. One limitation of the exposure vessels used was that they did not allow for any air exchange or the injection of ambient carbon dioxide, which both are essential for the algal cells to propagate.
Test duration must be 96 h.	Test duration was 96 h.

ASci Corporation/ASci-Duluth
Environmental Testing Division
ASCI Report ID# 003-AL01.RJM
ASCI Study ID# 5000-003-08

Appendix A

Chemical Analysis of Deionized Water

Chemical Analysis of Deionized Water^a

Parameter	µg/L	MDL ^b (µg/L)	Parameter	µg/L	MDL ^b (µg/L)	Parameter	Unit	Conc.
Alirin	ND ^c	0.3	Naicd	ND	2.3	Total Suspended Solids	mg/L	< 4
A-BHC	ND	3.0	Deoxione	ND	1.0	Ammonia Nitrogen	mg/L	< 0.05
B-BHC	ND	0.4	Ronari	ND	0.5	Total Kjeldahl Nitrogen	mg/L	< 0.05
D-BHC	ND	4.0	Chlorpyrifos	ND	0.3	Chemical Oxygen Demand	mg/L	5
Chlorfenc (Gamma)	ND	1.0	DEF	ND	0.3	Total Cyanide	mg/L	< 0.01
Chlorfenc (Alpha)	ND	1.0	Rotator	ND	0.3	Aluminum	µg/L	< 100
4,4'DDD	ND	0.3	Phosalone	ND	0.3	Arsenic	µg/L	< 2
4,4'DDE	ND	0.3	Oution	ND	5.0	Cadmium	µg/L	< 0.3
4,4'DDT	ND	0.3	Conamphos	ND	5.0	Calcium	mg/L	< 0.3
Dieldrin	ND	0.3	Dichlorvos	ND	1.0	Cobalt	µg/L	< 2
Endosulfan I	ND	1.0	Mevinphos	ND	3.5	Chromium	µg/L	< 2
Endosulfan II	ND	1.0	Trifluralin	ND	0.3	Copper	µg/L	< 1
Endosulfan Sulfate	ND	1.0	Ethoxypr	ND	0.5	Iron	µg/L	< 3
Endrin	ND	1.0	Phorate	ND	0.3	Lead	µg/L	< 2
Endrin Methylene	ND	0.3	Disulfoton	ND	0.3	Magnesium	mg/L	< 0.1
Heptachlor	ND	0.05	Methyl Parathion	ND	0.3	Mercury	µg/L	< 0.2
Heptachlor Epoxide	ND	0.3	Merphos	ND	0.5	Nickel	µg/L	< 2
Lindane (G-BHC)	ND	0.3	Feuthion	ND	0.3	Potassium	mg/L	< 0.3
Texaphene	ND	2.0	Diphenoamid	ND	0.3	Selenium	µg/L	< 1
Methoxychlor	ND	1.0	Ethion	ND	0.3	Silver	µg/L	< 1
Endrin Ketone	ND	1.0	Phenothio	ND	1.0	Sodium	mg/L	< 0.3
PCB 1016	ND	1.0	Carbophenox	ND	1.0	Zinc	µg/L	< 1
PCB 1221	ND	1.0	Diazinon	ND	0.3			
PCB 1237	ND	1.0	Dimethoate	ND	0.3			
PCB 1242	ND	1.0	Malathion	ND	2.0			
PCB 1248	ND	1.0	Parathion	ND	0.3			
PCB 1254	ND	1.0	Methyl Trithion	ND	1.0			
PCB 1260	ND	1.0	Prometon	ND	0.3			
			Trichloronal	ND	0.3			

^a The Water Sample Was Collected at Testing Facility and Analyzed During August-September 1991

^b MDL - Method Detection Limit

^c ND - Not Detected

Appendix B*

Isooctyl Acrylate: Method Validation for Analysis from Water

*A certified copy of the Method Validation report resulting from the ASCI Study ID# 3030-003-06 is appended. The report contains a total of 27 pages which are numbered consecutively from p. 1 - p. 27 and also re-numbered as "Page ____ of ____". These 27 pages are a part of the total number of pages included in this report.

Sponsor: 3M Company
Sponsor Study ID# 11774

STUDY TITLE

ISOOCTYL ACRYLATE: METHOD VALIDATION FOR ANALYSIS FROM WATER

AUTHORS

Minren Xu and Dinesh Vaishnav

STUDY COMPLETED

May 28, 1992

TESTING FACILITY

ASCI Corporation
ASCI-Duluth Environmental Testing Division
112 East Second Street
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STUDY IDENTIFICATION NUMBERS

ASCI Study ID# 5030-003-01

3M Company Study ID# J2774

CERTIFIED COPY

Signature: [Signature] Date: 5/27/92

Page 1 of 27

Sponsor: 3M Company
Sponsor Study ID# J2774

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CERTIFICATION OF GOOD LABORATORY PRACTICE COMPLIANCE

To the best of my knowledge, this study was conducted in accordance with OECD Good Laboratory Practice Standards (OECD Council Decision C(81)30, Annex 2: OECD Principles of Good Laboratory Practice 1981).

Study Director: _____ Date: _____
Minren Xu
ASCI Corporation/ASCI-Duluth
Environmental Testing Division

Based on the signatures of the Study Director and the Quality Assurance Auditor, this study, to the best of our knowledge, was conducted in accordance with OECD Good Laboratory Practice Standards (OECD Council Decision C(81)30, Annex 2: OECD Principles of Good Laboratory Practice 1981).

Sponsor: _____ Date: _____
Submitter: _____ Date: _____

STATEMENT OF QUALITY ASSURANCE

The study data were reviewed by the ASCI-Duluth Environmental Testing Division Quality Assurance Unit to assure that standard operating procedures and guidelines used to conduct this study were followed, and this report is an accurate reflection of the raw data. The types of audits performed are listed in the following table.

Type of Audit for ASCI Study ID# 5030-003-01	Audit Date	Date Reported to Study Director and Management
Study Plan	12-17-1991	12-17-1991
In-Life Phase	12-19-1991	12-19-1991
Raw Data and Draft Report	01-09-1992	01-09-1992
Final Report	05-28-1992	05-28-1992

Alan Mozol
Acting Manager, Quality Assurance Unit

Date: _____

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STUDY SUMMARY TABLE

Study Title	Isooctyl Acrylate: Method Validation for Analysis from Water
Good Laboratory Practice Standards	As promulgated under the OECD Council Decision C(81)30, Annex 2: OECD Principles of Good Laboratory Practice (OECD 1981).
Sponsor	Rich Purdy, 3M Environmental Laboratory, Building 2-3E-09, 935 Bush Avenue, St. Paul, MN 55106; Tel No. (612) 778-5379.
Sponsor's Representative	Susan A. Beach, 3M Environmental Laboratory, Building 2-3E-09, 935 Bush Avenue, St. Paul, MN 55106; Tel No. (612) 778-7452.
Testing Facility	ASCI Corporation/ASCI-Duluth Environmental Testing Division, 112 East Second Street, Duluth, MN 55805; Tel. No. (218) 722-4040.
Study Director	Minren Xu
Acting QAU Manager	Alan Mozol
Testing Facility Director	Donald Mount
Study Initiation Date	December 17, 1991
Test Dates	December 17-19, 1991
Test Substance	Isooctyl acrylate (CAS No. 29590-42-9, [MC-857, Lot 3290], 99.75% acrylate (as determined by Sponsor NB# 92391), liquid.

Test Description	Calibration Curves: (1) Standard solutions of various test substance concentrations and reagent (acetone) blank were prepared in acetone, (2) all solutions and reagent blank were analyzed twice by GC/MS, and (3) data were used to calculate regression equations, analytical method detection limits and other statistics.
Test Description (continued)	Spike Solutions and Recoveries: (1) Three replicates of test substance low and high spike solutions, and method blank (deionized water) were prepared using deionized water, (2) spike solutions and method blank were extracted using solid/liquid extraction technique, and extracts analyzed by GC/MS, and (4) data were used to calculate test substance recoveries from spike solutions.

Test Results	<p>Percentage relative standard deviation (% RSD): First calibration curve -- 0.81% Second calibration curve -- 1.93%</p> <p>Correlation coefficient (r): First calibration curve -- 1.000 Second calibration curve -- 0.999</p> <p>Method detection limit (MDL): With first calibration curve -- 0.04 mg/L With second calibration curve -- 0.04 mg/L</p> <p>Mean percentage recovery (R) from low spike solution (0.123 mg/L test substance): 85.91%</p> <p>Mean percentage recovery (R) from high spike solution (8.8 mg/L test substance): 103.48%</p> <p>Combined mean percentage (R) recovery from low and high spike solutions: 94.70%</p>
Location of Raw Data and Final Report	<p>ASCI Corporation/ASCI-Duluth Environmental Testing Division, 112 East Second Street, Duluth, MN 55805; Tel. No. (218) 722-4040.</p>

1.0 INTRODUCTION

The test substance, isooctyl acrylate, is an ester made from primarily isooctanol and acrylic acid. According to OECD recommendations for new chemical substances (OECD Council Decision, 12th May, 1981; C(81)30), (1) the test substance physical-chemical properties and toxicities to various aquatic organisms need to be determined, and (2) chemical effects must be reported on the basis of measured chemical concentration. For the latter, there was a need to validate an analytical method so that test substance concentration can be determined from matrices employed in various tests. The analytical method was provided by the Sponsor.

The objectives of the present study were: (1) to develop an acceptable calibration curve, (2) to calculate detection limit of the analytical method, and (3) to determine test substance recoveries from spike solutions prepared using deionized water.

2.0 TEST METHODS

2.1 Formulas and Definitions. The formulas and definitions used in this study were:

- (1) Test Substance Mean Percentage Recovery (R)

$R_i = (\text{Measured concentration} / \text{Target concentration}) \times 100$

The mean R was calculated using individual R_i values which fell within $R \pm 3SD$ range. If, the mean R was not between 80% and 120%, all measured concentrations were corrected accordingly.

(2) Method Detection Limit (MDL)

$MDL = 3 \times \text{background signal in reagent blank}$

(3) Relative Standard Deviation of Calibration Curve (% RSD)

$\% RSD = (\text{Standard deviation of slope} / \text{slope}) \times 100$

(4) The sample response was corrected for the response of the method blank, if interference from the method blank was expected to have any effect on the sample response.

2.2 Test Substance. The test substance, isooctyl acrylate, (CAS No. 29590-42-9 [] Lot 3290) was received at ASCI on October 3, 1991 in one amber glass bottle placed in a sealed metal container. The test substance was stored at room temperature as received. According to a material safety data sheet and a written communication provided by the Sponsor, (1) the test substance was a clear, colorless, mobile liquid with acrylate odor, (2) the test substance concentration in deionized water can be analyzed by a GC method, (3) the test substance was 99.75% acrylate as determined by Sponsor [] and (4) the test substance had 1 mm Hg vapor

Sponsor: JMI Company
Sponsor Study ID# 37714

pressure at 50°C. The Sponsor also had information that based on the chemical structure, there would be essentially no dissociation or pH-dependent hydrolysis of the test substance at environmental pH levels.

2.3 Apparatus and Reagents. The apparatus and reagents used were:

- (1) HP model 5890 gas chromatograph with 30 m 0.32 DB-5 (J & W Scientific) capillary column;
- (2) HP model 5970 mass spectrometer;
- (3) Pesticide grade methylene chloride and other solvents;
- (4) Deionized water; and
- (5) Extraction apparatus.

2.4 GC/MS Analysis. The analytical conditions were:

- (1) Carrier gas: Helium at a total inlet purge flow of 40 ml/minute and a septum purge flow of 1 ml/minute with splitless injection mode;
- (2) Temperature program: Isothermal at 70°C for 2 minutes then 8°C per minute to 200°C;
- (3) Ionization source: Electron impact with a scan range of 20-500 mμ; and
- (4) Detection method: Total ion chromatograph.

Before analysis, mass spectrometer was tuned using autotune program. A GC column performance test was conducted using column check sample (HP Sample A) to meet the criteria recommended by the manufacturer. A post GC/MS performance test was carried out by running a column check sample (HP Sample A) to ensure the stability of the instrument during the analytical test.

2.5 Calibration Curve. Two test substance stock solutions were prepared in acetone in 10-ml volumetric flasks. The first solution contained 1,760 mg/L test substance and the second solution contained 880 mg/L test substance. Subsequently, four standard solutions were prepared by adding appropriate volumes of the second stock solution to 10-ml volumetric flasks and diluting to volume with acetone. A reagent blank was prepared using acetone.

Each stock and standard solution, and reagent blank were analyzed twice by GC/MS. The instrument responses, except of reagent blank, from 8.95 to 12.958 minutes were integrated using a group integration method, and correlated with the test substance nominal concentration. The relative standard deviations of calibration curves (% RSD) and method detection limits (MDL) were then calculated.

2.6 Spike Solutions. Three replicates of a low level spike solution were prepared by adding 7 μ l of test substance second stock solution (880 mg/L) to 50 ml of deionized water. This produced a target spike concentration of 0.123 mg/L test substance. Similarly, three replicates of a high level spike solution were prepared by adding 5 ml of test substance second stock solution (880 mg/L) to 500 ml of deionized water. This produced a target spike concentration of 8.8 mg/L test substance. A method blank was prepared using 500 ml of deionized water.

2.7 Test substance Extraction and Analysis. Both spike solutions and method blank were first extracted, using solid/liquid extraction procedure, and extracts analyzed by GC/MS. The extraction procedure was:

- (1) Placed a 25-mm (with 50 ml sample) or 47-mm diameter (with > 50 ml sample) Empore[®] extraction disk (J.T. Baker, Inc.) between a filter base and reservoir;
 - (2) Pre-washed the disk with 10 ml of methylene chloride (elution solvent);
 - (3) Applied vacuum to draw the solvent through the disk;
 - (4) Added 10 ml of methanol, applied vacuum and left a meniscus of methanol just above the top of the disk
- (NOTES: RELEASED VACUUM BEFORE THE DISK WAS DRY. DID NOT

ALLOW DISK TO DRY AT ANY TIME BEFORE SAMPLE FILTRATION WAS COMPLETED);

- (5) Added 20 ml of deionized water to the reservoir, applied vacuum and left a meniscus of water just above the top of the disk;
- (6) Added 5 ml methanol per liter of sample and mixed well;
- (7) Poured sample into the reservoir and applied vacuum. The minimum filtration time was 10 minutes/L of sample;
- (8) After the sample was processed, drew air through disk for 15 minutes;
- (9) Placed the tip of the filter base into a test tube inside the filtration flask;
- (10) Rinsed the volumetric flask with 2.5 ml (with 50 ml sample) or 4-5 ml (with > 50 sample) methylene chloride and added the solvent to the reservoir;
- (11) Drew half the solvent through the disk and let stand for approximately 1 minute. Drew the remainder through the disk;
- (12) Repeated Steps 10 and 11 three times;
- (13) Collected a measured volume of methylene chloride extract; and
- (14) Processed the method blank in the same way (Steps 1 to 13) as the sample.

For low spike solutions, extracts were first concentrated under a gentle stream of nitrogen gas and the volumes of concentrated extracts measured. The extracts of both low and high spike solutions were then transferred to analytical vials and analyzed for the test substance concentrations using the GC/MS instrument. The instrument was operated as per manufacturer's recommendation.

2.8 Test Substance Recovery. The instrument responses between 8.95 and 12.958 minutes were integrated using a group integration method, and fitted to the first calibration curve to determine test substance concentrations. These data were then used to calculate the test substance percentage recoveries from spike solutions.

2.9 Test Substance Analysis During Various Tests. Several physical/chemical and toxicity tests were performed separately with this test substance. In analyzing the test substance concentrations in aqueous samples from these tests, the following procedure was used:

- (1) At each test initiation, developed an acceptable new calibration curve with a relative standard deviation (\pm RSD) within 10%;

- (2) Each day when test substance concentrations in aqueous samples from a particular test were analyzed, re-validated the previous calibration curve (from Step 1) using at least two standard solutions, or developed a new acceptable calibration curve with a relative standard deviation (% RSD) within 10%. In case of re-validation, the previous calibration curve was considered valid and the same regression equation (From Step 1) was used, if the measured and nominal concentrations of standard solutions did not differ by more than 10%;
- (3) Each time when test substance concentrations in aqueous samples from a particular test were analyzed, standard (deionized water) and test (e.g. well water, algal medium etc.) matrices blanks, and spiked standard and test matrices were prepared. The test substance spike concentration was close to the lowest nominal concentration used in a particular test. Generally, the spike concentrations were similar to the low spike concentration (0.123 mg/L) used in this method validation study;
- (4) Analyzed both standard and test matrices and calculated percentage spike recoveries;

- (5) Accepted spike recoveries if they were within the same range ($85.91 \pm 22.859\%$) as low spike recovery established from this method validation study;
- (6) Each time when test substance concentrations in aqueous samples from a particular test were analyzed, corrected (to 100%) test substance concentrations in aqueous samples for the percentage matrix spike recovery for that time.

2.10 Data Analysis. All data were analyzed using Minitab[®] statistical software (Minitab, Inc. 1988), MS ChemStation software (HP 1990) which interfaced the GC/MS instrument, and a scientific calculator.

3.0 RESULTS

Six test substance solutions, including two stock and four standard solutions (Table 1), were used to prepare two calibration curves. The use of a broad range of solution concentrations was important because the test substance concentrations in biological tests are expected to range from approximately 0.1 mg/L to the test substance water solubility concentration (12.44 mg/L).

The samples from physical/chemical and biological tests will be extracted and test substance concentrations eluted in approximately 15 ml of solvent (actual extract volume will be measured). Accordingly, one solution (standard solution 1) used for the two calibration curves had a test substance concentration approximately 3 fold greater than the method detection limit (MDL) of 0.04 mg/L (Table 1). All other solutions, except the first stock solution, were below and near the test substance solubility (12.44 mg/L) in deionized water (Table 1). The test substance concentration in the first stock solution was approximately twice the solubility concentration.

The GC/MS responses in two calibration curves are listed in Table 2. Correlations of GC/MS response (ordinate) and test substance nominal concentration (abscissa) had correlation coefficients (r) of 1.000 and 0.999 for the first and second calibration curves, respectively (Table 3). The slopes from both curves differed by approximately 0.32%, and relative standard deviations (% RSD) of slopes were 0.81% and 1.93% for the first and second calibration curves, respectively (Table 3). The detection limit of 0.04 mg/L test substance was the same as calculated for both calibration curves (Table 3).

The low spike concentration was 0.123 mg/L test substance and high level spike concentration was 8.8 mg/L test substance (Table 4). These concentrations were within the range of test substance concentrations to be used in biological and physical/chemical tests. The volumes of spike solutions (50 ml and 500 ml) used were comparable to the volumes that may be analyzed from physical/chemical and biological studies. The test substance recoveries for the low spike solution ranged between 70.73% and 112.20% with a mean of $85.91 \pm 22.859\%$, and for the high spike solution between 97.50% and 111.36% with a mean of $103.48 \pm 7.121\%$. (Table 4). The combined mean recovery for low and high spike solutions was $94.70 \pm 17.943\%$ (Table 4).

The test substance concentration in the method blank was below the method detection limit of 0.04 mg/L isooctyl acrylate.

From the quality assurance standpoint, this test is acceptable because it complies with the acceptance criteria (Table 5).

4.0 CONCLUSIONS

The GC/MS response and test substance, isooctyl acrylate, concentrations between 8.8 and 1,760 mg/L were in linear

correlation. The test substance combined mean recovery (94.70%) from low and high spike solutions suggested that extraction and analytical procedures should be adequate for use with other aqueous samples.

5.0 DEVIATIONS FROM APPROVED ASCI STUDY PLAN

The deviations which occurred while conducting this study were:

- (1) HP model 5890 gas chromatograph and HP model 5970 mass spectrometer were used instead of HP model 5970 gas chromatograph and HP model 5890 mass spectrometer.
- (2) In GC/MS analysis, total inlet purge flow of helium gas was at 40 ml/minute and a septum purge flow was at 1 ml/minute, instead of helium at 5.5 ml/min and a septum purge flow of 5.8 ml/minute.
- (3) In GC/MS analysis, temperature program used was 70°C for 2 minutes and then 8°C/minute to 200°C, instead of 70°C for 2 minutes, and then 8°C/minute to 220°C and holding at 220°C for 2 minutes, or as appropriate. This was because after 180°C nothing eluted from the GC column.

To the best of our current scientific knowledge and understanding,
this deviation should have no effect on the results presented in
this report.

6.0 REPORT SIGNATURE

Study Director: _____ Date: _____
Minren Xu
ASCI Corporation/ASCI-Duluth
Environmental Testing Division

7.0 REFERENCES

Hewlett Packard (HP). 1990. HP 59940A MS ChemStation (HP-UX series) Handbook.

Minitab, Inc. 1988. Minitab Release 6.1. Minitab, Inc., State College, PA.

Organization for Economic Cooperation and Development (OECD). 1981. OECD Guidelines for Testing of Chemicals. OECD Publication Information Center, Washington, DC.

8.0 PERSONNEL INVOLVED IN STUDY AND THEIR RESPONSIBILITIES

Personnel	Responsibility
Minren Xu	Study Director
Connie Coleson	Glassware preparation
Billie Samson	Laboratory assistance
Dinesh Vaishnav	Report preparation
Alan Mozol	QAU
Nancy Jordan	Archivist

Table 1. Isooctyl acrylate (test substance): Solutions for two calibration curves

Test substance solution	Dilution	Test substance nominal concn (mg/L)
Reagent blank	0.0 μ l test substance in 10 ml acetone (final volume)	0.0
First stock solution	20 μ l test substance in 10 ml acetone (final volume)	1,760
Second stock solution (SS)	25 μ l test substance in 25 ml acetone (final volume)	880
Standard solution 1	100 μ l SS in 10 ml acetone (final volume)	8.8
Standard solution 2	500 μ l SS in 10 ml acetone (final volume)	44
Standard solution 3	1,000 μ l SS in 10 ml acetone (final volume)	88
Standard solution 4	5 ml SS in 10 ml acetone (final volume)	440

Table 2. Isooctyl acrylate (test substance): GC/MS responses in two calibration curves

Test substance nominal concn (mg/L)	GC/MS response in first calibration curve	GC/MS response in second calibration curve
Reagent blank	19,622	19,622
1,760	2,719,832,005	2,729,584,720
880	1,390,089,059	1,258,512,351
8.8	22,481,557	10,280,168
44	62,891,391	52,827,478
88	128,917,851	113,808,095
440	658,002,779	622,643,636

Table 3. Isooctyl acrylate (test substance): Statistical analysis of two calibration curves^a

Parameter	First calibration curve	Second calibration curve
Regression equation	$-1.76e+06 + 1.55e+06 (x)^b$	$-2.48e+07 + 1.54e+06 (x)^b$
Slope \pm SD	1551104 \pm 12498 ^c	1546151 \pm 29918 ^c
Relative standard deviation (% RSD) ^d	0.81%	1.93%
Correlation coefficient (r)	1.000	0.999
Method detection limit (MDL) ^e	0.04 mg/L	0.04 mg/L

^aGC/MS response and isooctyl acrylate (test substance) concentration (milligrams per liter) were plotted on ordinate and abscissa, respectively.

^bEquation was generated using MS ChemStation software (HP 1990).

^cSlope and SD were calculated using Minitab[®] statistical software (Minitab, Inc. 1988), as HP-UX software did not calculate SD.

^dPercentage RSD = (Standard deviation of slope/slope) \times 100.

^eMDL = 3 \times response in reagent blank (= 19,622; Table 2)/slope.

Table 4. Isooctyl acrylate (test substance): Recoveries from spiked deionized water

Type of solution	Rep	Test substance target concn (mg/L)	Test substance measured concn (mg/L)*	% Recovery (R) ^b	Mean \pm SD% recovery (R) ^c
Method blank	1	0.0	<0.04 ^d	-	-
Low spike	1	0.123	0.092	74.80	85.91 \pm 22.859
	2	0.123	0.138	112.20	
	3	0.123	0.087	70.73	
High spike	1	8.8	8.58	97.50	103.48 \pm 7.121
	2	8.8	8.94	101.59	
	3	8.8	9.80	111.36	
Combined recovery from low spikes + high spikes					94.70 \pm 17.943

*Determined using first calibration curve (Table 3).

^bR_i = (Measured concentration/Target concentration) X 100.

^cMean R was calculated using R_i values which fell within R \pm 3SD range.

^dMethod detection limit (MDL) was 0.04 mg/L isooctyl acrylate.

Table 5. Isooctyl acrylate (test substance): QA criteria and test acceptability

QA criterion	Results
Relative standard deviation of calibration curve (% RSD) must be within 10%	% RSD of first calibration curve was 0.81% and of second calibration curve was 1.93%
Post run standard response must be within 10% of the same standard analyzed at the beginning of the test	Responses from all peaks from post run standard differed by 5.95% compared to the beginning of the test

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Signature: *[Signature]* Date: 5/27/92